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PROCESS AND APPARATUS FOR THE
SEMICONTINUOUS EXTRACTION OF NICOTINE FROM TOBACCO

Field of the Invention

This invention relates to extraction procedures and is particularly directed to an improved process for the extraction of nicotine from tobacco.

Background of the Invention

Various processes have been proposed for the removal of nicotine from tobacco. Most of these processes, however, adversely affect the desirable flavor and aroma properties of the tobacco. Also, they are often complex and expensive to carry out.

United States Patent No. 4,153,063 (Roselius) discloses a process for removing nicotine from tobacco in which tobacco is contacted with an extraction solvent in a supercritical state. It discloses both a single step extraction process and a multi-step extraction process. In the single step extraction process, moist tobacco is extracted with a solvent in a supercritical state. Because aroma components are also removed along with nicotine in this single step extraction process, the multi-step process is suggested. In the first step, dry tobacco is extracted with a solvent in the supercritical state to remove the aroma components. In the second step, the tobacco is moistened and again extracted

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with a solvent in the supercritical state to remove nicotine. The nicotine is separated from the solvent by either evaporating the solvent, contacting the solvent in a separate vessel with an acid, or adsorbing the nicotine on an active carbon column. In the third step, the stored aroma components from the first step are redissolved in a supercritical solvent and returned to the tobacco. This multi-step extraction process is expensive and time consuming. In addition, the prolonged handling of the aroma components may adversely affect their properties.

Summary of the Invention

This invention provides an improved process for removing nicotine from tobacco. A nicotine-free solvent in the supercritical state is fed into a first end of an extraction system containing tobacco and a nicotine-rich solvent is discharged from a second end of the extraction system. Periodically a portion of extracted tobacco is discharged from the first end of the extraction system while a portion of an unextracted tobacco is charged to the second end of the extraction system.

This invention also provides an improved process for removing nicotine from an extraction solvent. A nicotine-containing solvent in the supercritical state is fed into the first end of an entrapment vessel containing a nicotine entrapment material while a nicotine-free supercritical solvent is withdrawn from the second end of the entrapment vessel. Periodically a portion of spent entrapment material is discharged from the first end of the entrapment vessel while a portion of fresh entrapment material is charged to the second end of the entrapment vessel.

In one embodiment of this invention, a plurality of tobacco extraction vessels is connected

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in series. These extraction vessels are in turn connected to an entrapment vessel or a plurality of entrapment vessels which are also connected in series. Tobacco is extracted with a solvent either in the supercritical state or in the liquid state by continuously passing the nicotine-free solvent through one end of the plurality of extraction vessels connected in series and discharging the nicotine-enriched solvent from the opposite end. Thereafter the solvent, enriched in nicotine, is passed through an entrapment vessel or a plurality of entrapment vessels, connected in series, to remove the nicotine. The solvent, depleted in nicotine, is then recycled to the extraction vessel or vessels to again extract nicotine. Removal and addition of an extraction vessel from the system, or removal and addition of an entrapment vessel from the system, to provide continuous extraction or entrapment is accomplished by valve adjustment.

It is an object of this invention to provide a process for extracting nicotine from tobacco which is more efficient, provides a faster cycle time and results in lower capital and operating costs.

It is another object of this invention to provide a process for extracting nicotine from tobacco which increases the concentration of nicotine in the solvent and decreases the amount of solvent required per unit of tobacco.

It is still another object of this invention to provide a process for extracting nicotine from tobacco which requires less extraction solvent and thereby results in less degradation and loss of the aroma producing components and consequently gives an improved tobacco product.

It is another object of this invention to provide a process for extracting nicotine from tobacco which increases the amount of nicotine loaded on the

nicotine entrapment material and significantly decreases the entrapment material to tobacco ratio.

It is a further object of this invention to provide a process for extracting nicotine from tobacco which results in a reduced CO_2 pressure drop, reduced tobacco bed compaction and a more favorable extraction bed geometry.

These and other objects and advantages of the invention may be seen in the following description.

Brief Description of the Drawing

FIG. 1 illustrates an apparatus for the semi-continuous extraction of nicotine from tobacco.

FIG. 2 illustrates an apparatus for the semi-continuous extraction of nicotine from tobacco and the semi-continuous entrapment of nicotine from the solvent.

Detailed Description of the Invention

An apparatus for the semi-continuous extraction of nicotine from tobacco is shown in FIG. 1. Extraction vessels 10 and 11 contain tobacco and are connected in series. The vessels as depicted are on stream (all radial or all axial flow) and are connected to entrapment vessel 15.

An extraction solvent is supplied to extraction vessel 10 which is connected to a pump 16. The pressure in the vessel is controlled by means of a fill pump (not shown) and the temperature is controlled by means of heat exchanger 17. The extraction solvent enters the top of extraction vessel 10, passes downwardly through the tobacco bed and leaves at the bottom of the vessel. In passing through the tobacco bed, the extraction solvent becomes enriched with nicotine from the tobacco. The solvent is then circulated to extraction vessel 11, again being introduced

from the top, and then passing downwardly and exiting at the bottom. After exiting extraction vessel 11, the solvent is circulated to entrapment vessel 15. The extraction solvent enters the top of the vessel and then passes downwardly exiting at the bottom. In passing through the vessel, the nicotine in the solvent becomes trapped on the entrapment material. The solvent, depleted of nicotine, is then returned into the cycle by recirculating it to extraction vessel 10.

Extraction vessels 12, 13 and 14 are off stream and are in the turn around cycle. In the turn around cycle, extraction solvent is vented from the extraction vessel, the extracted tobacco is unloaded, unextracted tobacco is loaded into the extraction vessel, and the extraction vessel is refilled with extraction solvent. Extraction vessels containing extracted tobacco are removed periodically from the end into which nicotine-free solvent is fed while simultaneously extraction vessels containing unextracted tobacco are added at the end from which nicotine-enriching solvent is discharged. Removal and addition of extraction vessels is accomplished by valve adjustment.

FIG. 2 illustrates an alternative embodiment wherein a plurality of extraction vessels, connected in series, is connected with a plurality of entrapment vessels, also connected in series. Extraction vessels 10 and 11 contain tobacco and are connected in series and are on stream (all radial or all axial flow). Extraction vessels 12, 13 and 14 are off stream and in the turn around cycle. Entrapment vessels 20 and 21 are connected in series and are on stream (all radial or all axial flow).

As described for FIG. 1, extraction solvent is supplied to extraction vessel 10 and then circulated to extraction vessel 11. After exiting extrac-

tion vessel 11, the solvent is circulated to entrapment vessel 20. The extraction solvent enters the top of the vessel and passes downwardly exiting at the bottom. The solvent is then circulated to entrapment vessel 21 again being introduced from the top, and passing downwardly exiting at the bottom. In passing through the vessels 20 and 21, the nicotine in the solvent becomes trapped on the entrapment material. The solvent, depleted of nicotine, is then returned into the cycle by recirculating it to extraction vessel 10.

Entrapment vessel 22 is off stream and in the turn around cycle. In the turn around cycle, extraction solvent is vented from the entrapment vessel, the spent entrapment material unloaded, fresh entrapment material is loaded into the vessel and the vessel is refilled with extraction solvent. Entrapment vessels containing spent entrapment material are removed periodically from the end in which nicotine-enriched solvent is fed while simultaneously entrapment vessels containing fresh entrapment material are added at the end from which nicotine-lean solvent is discharged. Removal and addition of entrapment vessels is accomplished by valve adjustment.

In yet another embodiment of this invention, a plurality of entrapment vessels connected in series may be used to remove nicotine from a solvent in a process utilizing a single extraction vessel rather than a plurality of extraction vessels connected in series.

A number of extraction solvents having solvent capacity for nicotine in both their liquid and gaseous phases can be employed to reduce the nicotine content of tobacco. Carbon dioxide in the supercritical state is the preferred solvent in this invention. Other useful solvents include, for

example, halogenated hydrocarbons including up to about 4 carbon atoms such as CF_4, CHF_3CClF_3, $CBrF_3$, $CF_2{=}CH_2$, $CF_3{-}CF_2CF_3$, $CHClF_2$, CCl_2F_2, $CHCl_2F$, CCl_3F, $CBrF_3$, $CFCl{=}CF_2$, $CH_3{-}CF_3$, octafluorocyclobutane and hydrocarbons including up to about 5 carbon atoms such as propane, butane, pentane; other useful solvents include N_2O, SF_6 and argon. Mixtures of solvents or additives or co-solvents may be used to obtain improved solvent characteristics.

Supercritical carbon dioxide is carbon dioxide which is above its critical temperature, i.e., above 31.3°C. and above its critical pressure, i.e., above about 70 atmospheres. Extraction with carbon dioxide in the supercritical state is carried out at a pressure in the range of from about 70 to about 1500 amospheres and at a temperature in the range of from above the critical temperature to about 120°C. The range of temperature and pressure for the supercritical state of other useful solvents are of generally the same order of magnitude.

The entrapment material in the entrapment vessel may be an adsorbent with an affinity for nicotine. Such adsorbents include activated carbon, silica, alumina, magnesium silicate and ion exchange resins. The adsorbent may also be mixed with a diatomaceous earth, up to a ratio of about 1:1, to improve the flow rate of the adsorbent.

Alternatively, the entrapment material in the entrapment vessel may be an absorbent which has an affinity for nicotine. Absorbents are preferred over adsorbents. Such absorbents include water, acid, aqueous acid solutions and aqueous salt solutions.

The preferred acids for use as an entrapment material in this invention are non-volatile and non-soluble in the solvent under the conditions of the extraction. Useful acids are sulfuric, phosphoric and nitric. Other useful acids are the polycarboxylic

acids such as tartaric, citric, malic, lactic, malonic, succinic, acetic and glutamic.

Monovalent salts of the above acids such as the alkali salts are generally preferred because these salts are less volatile and less soluble in the solvent. A preferred salt of an acid is mono-potassium citric acid. Monoammonium and diammonium salts of the above acids may also be used. Polyvalent salts of the above acids are also useful but are less efficient in trapping nicotine.

The extraction process may be carried out on tobacco which has or has not been premoistened. It is generally preferred to moisten the tobacco up to about 25% OV (oven volatiles). The percentage of oven volatiles (% OV) in the tobacco is a measure of the moisture content plus a minor fraction of other components and is determined as follows:

$$\% \text{ OV} = \frac{\text{weight loss of sample after 3 hrs. at } 100°\text{C}}{\text{sample weight}}$$

In a typical semi-continuous extraction and entrapment, as illustrated in FIG. 2, the cycle time for extraction vessel 10 was 1 hour and the total extraction was 2 hours. Turn around time for the extraction vessel was 3 hours. Cycle time for entrapment vessel 15 was 3 hours and the total entrapment (adsorption/absorption) time was 6 hours. Turn around time for an entrapment vessel was 3 hours.

Table 1 shows a comparison of nicotine concentration in supercritical carbon dioxide in a batch extraction versus a semicontinuous extraction. The amount of nicotine removed from the tobacco is shown, on a dry weight basis (dwb). The average nicotine concentration in the carbon dioxide increased from 60 PPM in a batch extraction to 107 PPM in a semi-continuous extraction which resulted in a decrease in the carbon dioxide/tobacco ratio from 300:1 to 168:1.

Table 1

COMPARISON OF
BATCH vs. SEMICONTINUOUS EXTRACTION

EXTRACTION TIME (hrs)	NICOTINE IN TOBACCO (%)	NICOTINE REMOVED, (dwb) (g/100g)	NICOTINE CONC. IN CO_2 (ppm)	
			BATCH	SEMI-CONTINUOUS
0	100	0		
1.0	86.8	1.605	107	107
2.0	2.7	0.195	13	107
		1.80 (total)	60 (avg)	107 (avg)

Table 2 shows a comparison of the carbon to tobacco ratio, on a dry weight basis (dwb), in a batch extraction-entrapment, a batch extraction-semicontinuous entrapment and a semicontinuous extraction-entrapment. The carbon to tobacco ratio dropped from 4.0 in a batch extraction-entrapment to 2.0 in a semi-continuous entrapment, which represented a decrease of 50% in the carbon to tobacco ratio. The estimated carbon to tobacco ratio in the semi-continuous extraction and entrapment was significantly below 2.0 (in the range of 0.4 to 0.8) which even when calculated from 0.8 still represents a decrease of 80% in the carbon to tobacco ratio when compared with the batch extraction-entrapment and a decrease of 60% when compared with the semi-continuous extraction.

Table 2

	Carbon/Tobacco Ratio (dwb)
Batch Extraction/ Batch Entrapment	4
Batch Extraction/ Semicontinuous Entrapment	2
Semicontinuous Extraction/ Semicontinuous Entrapment	less than 2 (0.4 - 0.8 (estimated))

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Table 3 shows a comparison of design features between a batch extraction and a semi-continuous extraction and entrapment. Full flavor tobacco having a nicotine content of 1.85% (dwb) and an oven volatile (OV) content of 25% is shown extracted with carbon dioxide under supercritical conditions at 260 atmospheres and 70°C using carbon as the entrapment material. After extraction, the tobacco had a nicotine content of 0.05%.

The number and size of the vessels required in a semi-continuous system are smaller than in a batch system resulting in reduced turn around time. In addition, the number of CO_2 circulation pumps, the size of the CO_2 storage vessels and the size of the CO_2 handling system (fill pump, compressor) are smaller. Only one heat exchanger, one recovery cooler-condensor and one dust filter are needed. The average nicotine concentration in the supercritical CO_2 was higher, which resulted in a lower value for the CO_2 to tobacco ratio and a lower value for the entrapment material to tobacco ratio. In addition, because the system runs semi-continuously, equipment reliability is better and adaptability of the system to a liquid wash process is easier.

Table 3

	BATCH EXTRACTION/ SEMICONTINUOUS ENTRAPMENT (4 UNITS)	SEMI-CONTINUOUS EXTRACTION/ ENTRAPMENT (4 UNIT EQUIV.)
cycle time	5 hrs	1 hr. extraction, 3 hrs. adsorption
extraction time	2 hrs	2 hrs
turn around time	3 hrs	3 hrs
CO_2/tob. ratio	300	168
carbon/tob. ratio	2	≤ 2
CO_2 flow	0.681 ~~m/m~~ MM lbs/hr per unit or 2.724 ~~m/m~~ lbs/hr total for 4 units	0.611 ~~m/m~~ lbs/hr total
EXTRACTION VESSELS:		
NO. OF VESSELS	4	5
TOTAL TOB. CHARGE, LBS. (dwb)	18,168	18,168
PER VESSEL TOB. CHARGE, LBS. (dwb)	4,542	3,634
PER VESSEL TOB. VOLUME, FT^3	458	366
NOMINAL VESSEL VOLUME, FT^3	2,765	640
NOMINAL VOLUME SIZE, FT	8D X 55H	4.5D X 40H
ENTRAPMENT VESSELS:		
NO. OF VESSELS	8	3
TOTAL CARBON CHARGE, LBS.	72,672	<65,600
PER VESSEL CARBON CHARGE, LBS.	9,084	<21,870
PER VESSEL CARBON VOLUME, FT^3	364	< 875
NOMINAL VESSEL VOLUME, FT^3	804	TBD
NICOTINE:		
LOADING ON CARBON, %	0.9	0.9+
LBS. REMOVED/CHARGE	328	65.6
CO_2 FLOW $\bar{M}$ LBS/HR	2.724	0.611
AVE. NICO. CONC. IN CO_2	60	107
M CO_2/M TOB. RATIO	300	168
TOTAL NO. OF PRESS. VESSELS	12	8

The following example is illustrative.

Eight pressure vessels were arranged for series extraction as shown in FIG. 2. The extraction vessels (10 through 14) were large enough to hold 5 4,849 lbs. of tobacco each at 25%OV (i.e., 366 cu.ft. of tobacco). The entrapment vessels (20 through 22)

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were large enough to hold 21,870 lbs. of activated carbon (i.e., 875 cu.ft. of carbon volume).

Dry carbon (21,870 lbs.) was loaded into each entrapment vessel (20, 21, 22). A blend of full flavor American tobacco was moistened from 12%OV to 25%OV by direct spray of deionized water in a rotating cylinder. About 4,845 lbs. of premoistened tobacco (i.e., 3,634 lbs. dwb) was loaded into each extraction vessel (10, 11, 12, 13, 14).

Start-Up

A CO_2 fill pump was used to pressurize vessels 10 and 20. Carbon dioxide was circulated through the two vessels at the rate of 611,000 lbs/hr. After extraction conditions were reached, i.e., 260 atmospheres, 70°C, the circulation of CO_2 was continued through vessels 10 and 20 for one hour. Vessels 11 and 21 were pressurized with CO_2 during this first hour.

In the second hour of extraction, the flow of CO_2 was directed through vessels 10, 11, 20 and 21 as shown in FIG. 2. Vessel 12 was pressurized with CO_2 during this second hour.

In the third hour of operation, the flow of CO_2 was directed through vessels 11, 12, 20 and 21.

Also in the third hour of operation, extraction vessel 10 was removed from the extraction loop and subjected to the turn-around phase. The CO_2 in vessel 10 was vented and the extracted tobacco was unloaded. Vessel 10 was again filled with tobacco and CO_2 and was ready to be returned to the extraction loop. The turn-around phase for an extraction vessel takes 3 hours.

In the fourth hour of operation, vessel 13 was added to the extraction loop and vessel 11 was removed and was subjected to the turn around phase.

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Also in the fourth hour of operation, the flow of CO_2 was directed through entrapment vessels 21 and 22. Entrapment vessel 20 was subjected to the turn-around phase. The CO_2 in vessel 20 was vented and the spent carbon was unloaded. Vessel 20 was again filled with carbon and CO_2 and was ready to be returned into the extraction loop. The turn-around phase for an entrapment vessel takes 3 hours.

Steady state conditions were reached in six hours. Vessel 10 was returned to the extraction loop in the 6th hour of operation (Table 4). Extraction vessels 11, 12, 13 and 14 were each similarly subjected to the turn-around phase and then returned to the extraction loop.

Continuous Operation

As shown in FIG. 2 and Table 4, one batch of extracted tobacco was produced every hour. The nicotine content in the tobacco was reduced 97%, from 1.85% nicotine (dwb) to 0.05% nicotine (dwb). The total extraction time was two hours. The solvent to tobacco ratio was 168 parts of CO_2 to one part of tobacco (dwb). This solvent to tobacco ratio was significantly lower than the 300 parts of CO_2 to one part of tobacco (dwb) required in a batch system.

As shown in Table 5, it was found that the continuous operation increases the concentration of nicotine in the CO_2 solvent without significantly increasing the concentration of other tobacco soluble materials (considered to be important for product quality) in the CO_2 solvent.

Expert evaluation showed that cigarettes made from extracted tobacco, where the solvent to tobacco ratio was low, were of higher subjective quality than cigarettes made from extracted tobacco where the solvent to tobacco ratio was high.

Table 4

	Time (Hr.)	CO_2 Flow Arrangement (1) Extraction Vessels	Entrapment Vessels	Vessels in Turn-around Phase (2)
Start-up:	1	10	20	
	2	10,11	20,21	
	3	11,12	20,21	10
	4	12,13	21,22	10,11,20
	5	13,14	21,22	10,11,12,20
	6	14,10	21,22	11,12,13,20
Continuous Steady State Operation:	7	10,11	22,20	12,13,14,21
	8	11,12	22,20	13,14,10,21
	9	12,13	22,20	14,10,11,21
	10	13,14	20,21	10,11,12,22
	11	14,10	20,21	11,12,13,22
	12	10,11	20,21	12,13,14,22
	13	11,12	21,22	13,14,10,20
	14	12,13	21,22	14,10,11,20
	15	----------Continous Operation-------------		

Note:

1 - CO_2 flow maintained at 611,000 lb/hr through vessels indicated.

2 - Turn-around time was 3 hours. Turn-around time (TAT) included the following steps.

CO_2 vent
Tobacco or activated carbon unloading
Tobacco or activated carbon loading
CO_2 fill to extraction conditions
(260 atmospheres, 70°C)

Table 5

	Batch Operation	Continuous Operation
% Nicotine in Tobacco (dwb)		
Unextracted	1.85	1.85
Extracted	0.05	0.05
Solvent/Tobacco Ratio (dwb)	300	168
Solubles in CO_2 (ppm)		
Nicotine	60	107
Other Tobacco Solubles	60-120	60-120
% Solubles Removed from Tobacco (dwb)		
Nicotine	1.8	1.8
Other Solubles	1.8-3.6	1.0-2.0

I claim:

1. A method for the solvent extraction of nicotine from tobacco which comprises:

(a) feeding a nicotine-free solvent to a first end of an extraction system containing tobacco and withdrawing a nicotine-rich solvent from a second end of the extraction system;

(b) periodically discharging a portion of extracted tobacco from the first end of the extraction system; and

(c) charging a portion of unextracted tobacco to the second end of the extraction system.

2. The method according to claim 1 wherein the solvent is in the supercritical state.

3. The method according to claim 1 wherein the solvent is in the liquid state.

4. The method according to claim 1 wherein the moisture content of the tobacco is up to about 30% by weight.

5. The method according to claim 1 wherein the extraction solvent is selected from the group consisting of carbon dioxide, argon, SF_6, N_2O, a lower hydrocarbon and a lower halogenated hydrocarbon.

6. The method according to claim 1 wherein the extraction process is carried out in a plurality of extraction vessels connected in series.

7. A method of separating nicotine from a solvent in the supercritical state which comprises:

(a) feeding a nicotine-containing supercritical solvent to a first end of an entrapment vessel containing a nicotine entrapment material and

withdrawing a nicotine-free supercritical solvent from a second end of the entrapment vessel;

(b) periodically discharging a portion of spent entrapment material from the first end of the entrapment vessel; and

(c) charging a portion of fresh entrapment material to the second end of the entrapment vessel.

8. The method according to claim 7 wherein the entrapment material is an adsorbent selected from the group consisting of carbon, silicon, alumina, magnesium silicate and ion exchange resins.

9. The method according to claim 7 wherein the entrapment material is an absorbent selected from the group consisting of water, acid, aqueous acid and aqueous salt solutions.

10. The method according to claim 9 wherein the entrapment material is monopotassium citric acid.

11. The method according to claim 7 wherein the entrapment process is carried out in a plurality of entrapment vessels connected in series.

12. A method for the extraction of nicotine from tobacco which comprises:

(a) feeding a nicotine-free supercritical solvent to a first end of an extraction vessel containing tobacco and withdrawing a nicotine-containing supercritical solvent from a second end of the extraction vessel;

(b) periodically discharging a portion of extracted tobacco from the first end of the extraction vessel;

(c) charging a portion of unextracted tobacco to the second end of the extraction vessel;

(d) feeding a nicotine-containing supercritical solvent to the first end of an entrapment vessel containing a nicotine entrapment material and withdrawing a nicotine-free supercritical solvent from the second end of the entrapment vessel;

(e) periodically discharging a portion of spent entrapment material from the first end of the entrapment vessel; and

(f) charging a portion of fresh entrapment material to the second end of the entrapment vessel.

13. The method according to claim 12 wherein the moisture content of the tobacco is up to about 30% by weight.

Clarify "lower"

14. The method according to claim 12 wherein the extraction solvent is selected from the group consisting of carbon dioxide, argon, SF_6, N_2O, a lower hydrocarbon and a lower halogenated hydrocarbon.

15. The method according to claim 12 wherein the entrapment material is selected from the group consisting of carbon, silicon, alumina, magnesium silicate and ion exchange resins.

16. The method according to claim 12 wherein the entrapment material is selected from the group consisting of water, acid, aqueous acid and acqueous salt solutions.

17. The method according to claim 16 wherein the entrapment material is monopotassium citric acid.

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(c) charging a portion of unextracted tobacco to the second end of the extraction vessel;

(d) feeding a nicotine-containing supercritical solvent to the first end of an entrapment vessel containing a nicotine entrapment material and withdrawing a nicotine-free supercritical solvent from the second end of the entrapment vessel;

(e) periodically discharging a portion of spent entrapment material from the first end of the entrapment vessel; and

(f) charging a portion of fresh entrapment material to the second end of the entrapment vessel.

13. The method according to claim 12 wherein the moisture content of the tobacco is up to about 30% by weight.

14. The method according to claim 12 wherein the extraction solvent is selected from the group consisting of carbon dioxide, argon, SF_6, N_2O, a lower hydrocarbon and a lower halogenated hydrocarbon.

15. The method according to claim 12 wherein the entrapment material is selected from the group consisting of carbon, silicon, alumina, magnesium silicate and ion exchange resins.

16. The method according to claim 12 wherein the entrapment material is selected from the group consisting of water, acid, aqueous acid and acqueous salt solutions.

17. The method according to claim 16 wherein the entrapment material is monopotassium citric acid.

18. The method according to claim 12 wherein the extraction process is carried out in a plurality of extraction vessels connected in series.

19. The method according to claim 12 wherein the entrapment process is carried out in a plurality of entrapment vessels connected in series.

20. An apparatus for the extraction of nicotine from tobacco with a solvent in the supercritical state or in the liquid state which comprises a plurality of extraction vessels connected in series.

21. The apparatus according to claim 20 wherein the extraction solvent is carbon dioxide.

22. An apparatus for the removal of nicotine from a solvent in the supercritical state or in the liquid state which comprises a plurality of entrapment vessels connected in series.

23. The apparatus according to claim 22 wherein the extraction solvent is carbon dioxide.

PROCESS AND APPARATUS FOR THE SEMICONTINUOUS EXTRACTION OF NICOTINE FROM TOBACCO

Abstract of the Disclosure

A process is provided for the improved removal of nicotine from tobacco. A nicotine-free solvent in the supercritical state is fed into a first end of an extraction system containing tobacco and a nicotine-rich solvent is discharged from a second end of the extraction system. Periodically a portion of extracted tobacco is discharged from the first end of the extraction system while simultaneously a portion of an unextracted tobacco is charged to the second end of the extraction system.

DECLARATION AND POWER OF ATTORNEY

I, Ravi Prasad, declare that I am a citizen of the United States of America, residing and having a post office address at 108-21 Henshaw Drive, Midlothian, Virginia 23110;

that I verily believe myself to be the original, first and sole inventor of the invention or discovery in:

PROCESS AND APPARATUS FOR THE SEMICONTINUOUS EXTRACTION OF NICOTINE FROM TOBACCO

which is described and claimed in the attached specification;

that I have read and do understand the content of said specification, including the claims, and knowledge and duty to disclose information, of which I am aware, which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a);

that I do not know and do not believe that this invention or discovery was ever known or used in the United States of America before my invention or discovery thereof, or patented or described in any printed publication in any country before my invention or discovery thereof, or more than one year prior to this application; or in public use or on sale in the United States of America more than one year prior to this application; that this invention or discovery has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months prior to this application; and that no application for patent or inventor's

certificate on this invention or discovery has been filed in any country foreign to the United States of America by me or my legal representatives or assigns;

and I hereby appoint Arthur I. Palmer, Jr., Esq., Reg. No. 18,855, James E. Schardt, Esq., Reg. No. 23,033, Albert E. Fey, Esq., Reg. No. 19,018, ~~and~~ and Thomas C. Secrest, Esq., Reg. No. 30,145, W. Edward Bailey, Esq., Reg. No. 30,994, my attorneys, with power of substitution, and with power of appointment of associate attorneys, and of revocation of their powers, to prosecute this application and any divisions, continuations in whole or in part, renewals and reissues of the same, and to transact all business in the Patent and Trademark Office connected therewith;

and I request that communications be sent to:

W. Edward Bailey
c/o Fish & Neave
875 Third Avenue
New York, New York 10022-6250

and that telephone calls be directed to:

W. Edward Bailey
(212) 715-0600.

Wherefore, I pray that Letters Patent be granted to me for the invention or discovery described and claimed in the attached specification and claims, and I hereby subscribe my name to the foregoing specification and claims, declaration, and power of attorney.

I declare, further, that I understand the English language and that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false

statements may jeopardize the validity of the application or any patent issuing thereon.

Date

Ravi Prasad

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